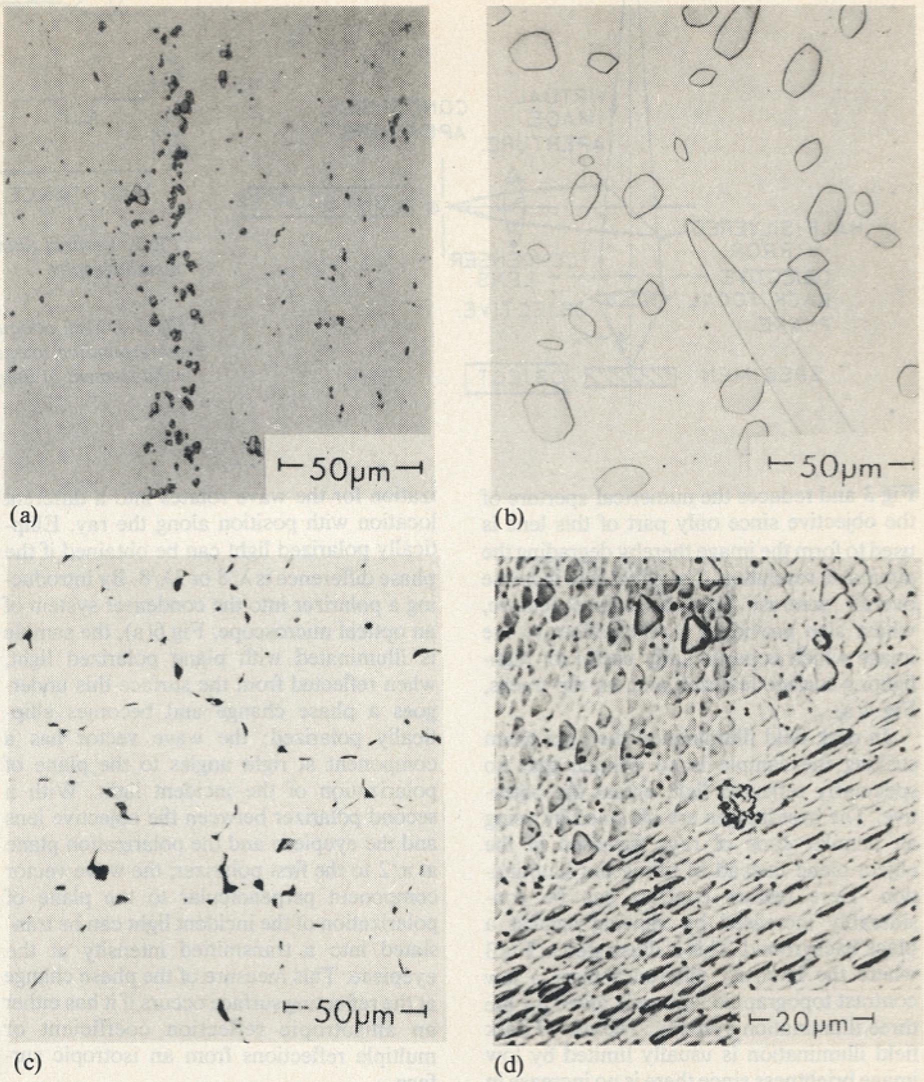
Optical Microscopy

*by P E J Flewitt and R K Wild**

The observation of polished and etched samples under an optical microscope remains the most useful and easily applied technique for establishing the microstructure of metals and alloys. However, as with any visual technique it depends critically upon the sampling procedure selected since the region viewed represents only a small fraction of the total volume of material. Since sample selection is such an important stage in any microstructural evaluation it must be undertaken to ensure that all necessary and appropriate information will be observed. Indeed it is often desirable to select samples not only from different regions of the whole but also three orthogonal sections.

Samples are prepared by a mechanical lapping sequence followed, in certain special circumstances, by final chemical or electrochemical polishing to remove the 'flowed' surface layer[1]. The contrast observed under the microscope then results from either an inherent difference in the light absorption characteristics of the phases present, 1(a) or it may be induced by preferential staining or attack of the surface by etching with a chemical reagent. A typical example of contrast arising from the latter is a polycrystalline copper 40 per cent zinc alloy etched with ferric chloride to reveal the Widmanstätten α rod-like precipitates in the β' phase matrix, 1(b). Here the quantity of light reflected into the objective of the optical microscope is determined by the orientation of these crystal planes within each phase. This dependence on contrast has resulted in a large bibliography describing the choice of etchants to produce appropriate images. For example it is possible to use selective etchants to identify a given phase; Fig 1(c) shows a 316 austenitic stainless steel which has been etched in picric acid to reveal the sigma phase precipitates positioned at the grain boundaries[2]. Etching can be taken a stage further to develop crystallographic shaped etch pits with faces parallel to low index lattice planes. Figure 1(d) shows etch pits in a niobium single crystal which has been subject to a tensile strain at room temperature. The three fold symmetry of the pits shows a $\{111\}$ orientation for the crystal and reveals that the region of the kink band has been subject to a local rotation of the crystal lattice. Therefore these etching features provide a means of examining crystal orientation[3].



(a) (b) (c) (d)

Fig 1. Optical micrographs showing:
(a) Stainless steel with inclusions of manganese sulphide and titanium carbo-nitride, (b) Copper-40% zinc alloy etched with ferric chloride to reveal the Widmanstätten α rod-like precipitates, (c) 316 austentic stainless steel etched with picric acid to selectively reveal the grain boundary σ phase[2], (d) A single crystal of niobium $[111]_t$ orientation deformed under uniaxial tension and etched with pitting reagent described by A G Vardiman and M R Achter (Trans Met Soc AIME, 1968, 242, 296) showing etch pits across a kink boundary[3].

Although image contrast is improved by etching to develop different local coefficients of scattering and reflection it does not always reveal all necessary detail. However, a number of purely optical methods of enhancing contrast applicable to any kind of surface condition are available including dark field and oblique illumination, polarized light, phase and interference contrast.

Vertical illumination

Vertical illumination is used for the examination of opaque samples by incident light, Fig 2. Here the reflected light from the vertical illumination is axial so that normal surfaces appear bright, whereas inclined surfaces such as pits and grain boundaries appear dark. Optimum illuminating conditions are achieved by eliminating unwanted light reflected from the numerous surfaces of the lens inside the microscope. Such background glare can be removed by the use of coated lenses and by inserting an iris diaphragm at the virtual image plane of the object in the condenser system. Further improvements in contrast can be obtained by providing critical illumination. This is achieved by adjusting the condenser lens system so that the image of the light source lies close to the plane of the sample ensuring the objective aperture is uniformly illuminated thereby making maximum use of the numerical aperture available.

Oblique and dark field illumination

A vertical illuminator can be tilted to secure either oblique or dark field illumination[4], Fig 3. Oblique illumination is obtained by displacing either the condenser lens system or the condenser aperture from the optical axis. This illuminates the sample from one side,

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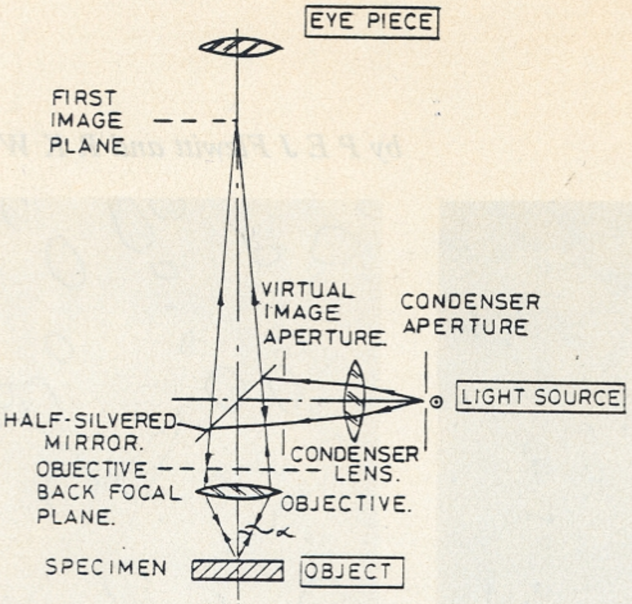


Fig 2. Vertical illumination light microscope used for routine metallography

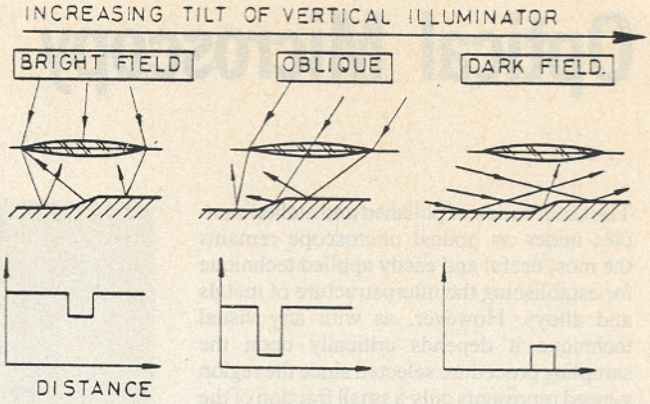


Fig 3. Direct, oblique and dark-field illumination together with corresponding image intensity distributions showing progressive enhancement of image feature.

Fig 3 and reduces the numerical aperture of the objective since only part of this lens is used to form the image thereby degrading the attainable resolution. The effect is to increase overall contrast from a surface feature, whilst also providing a relief effect to the image which is particularly useful for highlighting surface features such as slip traces, Fig 4(a).

In dark field illumination the light beam striking the sample is so oblique that no specularly reflected light enters the objective. The best images are obtained by using an annular cone of rays focussed in the object plane instead of tilting the illumination. Here feature contrast can be considerably increased by viewing against a black background. This is illustrated in Fig 3 where the intensity difference from a low contrast topographic feature is given for the three illumination systems. The use of dark field illumination is usually limited by low image brightness since there is no increase in the amount of information recorded in the image only an improvement in relative contrast. Figure 4(b) shows slip traces intersecting a second phase precipitate which has in itself imparted a tilt to the surface.

Polarizing illumination

A light wave oscillates perpendicular to the containing ray in all planes[5], Fig 5, and if the motion is sinusoidal it can be considered to be rotated through 360° about the ray to generate a three dimensional image and the phase is independent of the plane selected. However, when oscillation occurs in one plane the ray is defined as plane polarized. If two waves with the same phase oscillating in planes at right angles are combined this is equivalent to a third wave of the same phase but in a different plane. Thus, any plane polarized wave can be resolved into two components lying in arbitrary planes separated by $\pi/2$. Similarly, combining two waves, which are out of phase by $\lambda/4$ or $3\lambda/4$, produces a circularly polarized wave represented by a helix along the ray direction. Under such a condition the plane of polarization for the wave rotates into a different location with position along the ray. Elliptically polarized light can be obtained if the phase difference is $\lambda/8$ or $3\lambda/8$. By introducing a polarizer into the condenser system of an optical microscope, Fig 6(a), the sample is illuminated with plane polarized light, when reflected from the surface this undergoes a phase change and becomes elliptically polarized; the wave vector has a component at right angles to the plane of polarization of the incident light. With a second polarizer between the objective lens and the eyepiece and the polarization plane at $\pi/2$ to the first polarizer, the wave vector component perpendicular to the plane of polarization of the incident light can be translated into a transmitted intensity at the eyepiece. This measure of the phase change at the reflecting surface occurs if it has either an anisotropic reflection coefficient or multiple reflections from an isotropic surface.

Figure 6(b) shows a plastically deformed anisotropic metal, cadmium (hcp), where deformation twins are revealed by the polarizing illumination. Even for isotropic metals similar effects can be obtained by forming an anisotropic surface film, of thickness at least $\lambda/4$, whose orientation depends upon that of the substrate. Indeed, the thickness of oxide coatings on aluminium base alloys can be established using this procedure. In addition, polarized light can aid phase identification in alloy systems[6] where use is made of the behaviour on rotating a phase between crossed nicols in reflected light. For example sixteen of the phases commonly found in commercial aluminium alloys can be simply identified. Moreover, it is particularly helpful for the examination of non-metallic inclusions, graphite and silica in steels. In the case of inclusions it is possible to classify these on the basis of their opti-

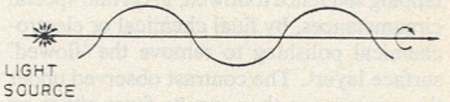


Fig 5. Light waves emitted from a source[5]

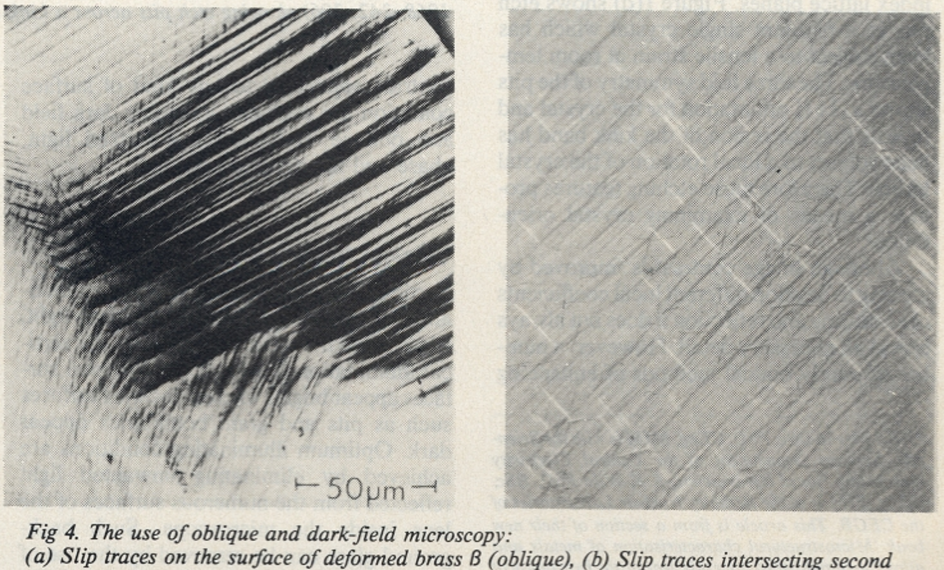


Fig 4. The use of oblique and dark-field microscopy:
(a) Slip traces on the surface of deformed brass β (oblique), (b) Slip traces intersecting second phase bainitic α_1 plates in a Cu-40% Zn alloy (dark-field).

cal properties and establish identity from response to polarized light[7].

Opaque stop microscopy

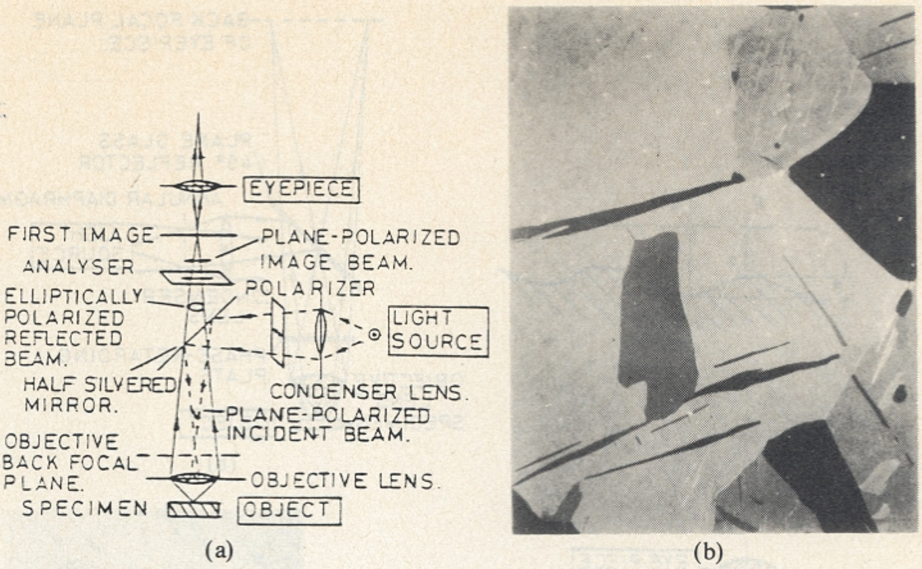
Figure 7(a) illustrates the principle of the opaque stop technique[4] where a ring stop (a metal disc with an annular opening) is positioned between the light source and the condenser. This forms a hollow cylinder of light focussed on the sample so that if the surface contains areas inclined at a small angle θ each will produce an image of the stop ring in the back focal plane, Fig 7(b). These will be displaced from each other by 2θ, however that part normal to the incident illumination will encounter the stop thereby limiting the image to the inclined surface.

Phase and interference contrast microscopy

Light reflected from a surface forms an image of the condenser aperture in the back focal plane of the objective lens, Fig 2. Hence a proportion of light passes outside the direct image of the condenser aperture. If the latter is an annulus, small differences in height on a sample surface change the path length of light reaching the image plane aperture giving a diminished intensity by the corresponding phase shift. Figure 8(a) considers a reflecting, dephasing and etched surface, where the optical path difference is $2t^*$ with respect to the reference plane and t^* is the height of perturbations. If the phase change on reflection at points P and Q is identical, then the phase difference ϕ is:

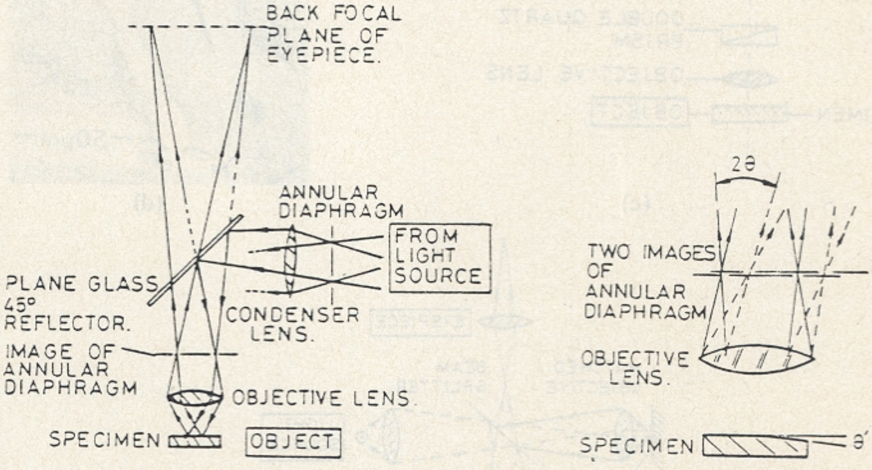
$$\phi = 4\pi t^*/\lambda$$

For phase detail to be visible the phase changes have to be converted into differences in light intensity (amplitude differences) which is achieved either qualitatively by phase contrast or quantitatively by interference contrast. The optical system used for phase contrast in the reflection microscope, Fig 8(b), includes phase plates made from magnesium fluoride which advance and retard the phase of the main beam. This allows features to be shown in direct or reverse contrast if the phase shift at the surface is small since the reflected beam is out of phase by $\pi/2$ with the main beam. Usually contrast is obtained from surface irregularities 20 to 50 nm high with a resolution limit of approximately 5 nm; however small differences in surface height are revealed rather than slope[8]. A development from phase contrast microscopy is the interference contrast technique described by Nomarski[9] which includes a double quartz wedge, Fig 8(c), in the optical system. When illuminated with polarizing light a double image of a specimen surface is obtained with a small lateral shift between the two images. Since the path length of the two beams is identical they can be made to interfere, using an analyser, wherever features of the two images are non-coincident. Figure 8(d) shows the relief developed on a prepolished surface as a consequence of ferrite precipitation; small differences in level are reproduced in high contrast.



(a) (b)

Fig 6. Polarizing microscopy:
(a) Optical microscopy fitted with crossed polars, (b) Deformed cadmium showing deformation twins.



(a) (b)

Fig 7. Opaque stop microscopy:
(a) Basic components of microscope, (b) Displacement of the image of the ring stop arising from a surface tilt.

Phase contrast arises from interference produced between light reflected from each region of a sample surface and that diffracted from the same region. Other interference techniques use light reflected from a second, reference, surface to interfere with light reflected from the sample producing either a two beam or multiple-beam interference pattern[10]. Alternatively the image of the second surface may be projected onto the plane of the specimen. Two beam interferometry is probably the most widely used of these techniques, Fig 8(e). Here monochromatic light reflected from the reference surface is out of phase with that reflected from the sample by an amount dependent on the difference in path length at the final image. Extinction occurs when the phase difference is π, that is when the difference in path length:

$$d = (2n + 1)\,\lambda/2\,\mu$$

where n is an integer, μ is the refractive index and λ the wavelength of the monochromatic light. Figure 8(f) shows a typical two beam interference micrograph which reveals the direction and magnitude of the surface displacements associated with twinning in a deformed Mo-Ru alloy. For monochromatic thallium illumination the fringe spacing $\lambda/2 = 270$ nm so that a step of height greater than $\lambda/2$ on the surface produces a displacement of more than one fringe spacing. Since all fringes appear identical only a fractional fringe displacement can be established easily. To identify a unique displacement the pattern has to be observed a second time using white light (tungsten) where fringes are dispersed into spectral colours allowing the number of integral fringe displacements to be established. The sensitivity to differences in height of the surface is the order $\lambda/20$ or about 25 nm. Compared with two beam interferometry, multi-beam techniques increase sensitivity a hundred fold allowing step heights of 0.5 nm to be measured. However, the latter techniques are less widely used due to many practical difficulties including the need to both coat the sample and reference flat with a thin partially transmitting, but largely reflecting film such as silver and use long working distance objectives to accommodate a reference flat positioned above the specimen surface.

Hot and cold stage microscopes

In addition to the range of optical microscopy techniques there is often a need to study the kinetics of several processes above and below ambient temperature in metals and alloys such as recrystallization and precipitation of a second phase. For these studies use is made of one of several hot[11] and cold[12] stage microscopes. As a consequence of geometrical constraints imposed by the need to either heat or cool the sample under a controlled atmosphere long working distance objective lens are usually used. These lens incorporate reflecting components with a normal objective lens which introduces a restriction to the value of the numerical aperture achievable. Despite restrictions these techniques have specific advantages, for example in the case of high temperature microscopy a precipitation process can be followed provided the newly formed precipitate can be viewed; if a surface relief is imparted to a prepolished surface. Fig 9 shows a sequence involving nucleation and growth of Widmanstätten α rods which precipitate during isothermal ageing of a metastable β' phase Cu–40%Zn alloy during heat treatment at 573 K. From such sequences recorded on film or video tape it is possible to establish the incubation period and then the lengthening kinetics of these α rods.

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References

1. L E Samuels, Metallographic Polishing by Mechanical Methods, Pitmans (London) 1968.
2. D J Chastell and P E J Flewitt, Mat Science and Eng 1979, **38**, 153.
3. P E J Flewitt and A G Crocker, Phil Mag 1976, **34**, 77.
4. D G Brandon, Modern Techniques in Metallography, Butterworths (London) 1966.
5. V A Philips, Optical Techniques for Metallographic Examination, in 'Techniques in Metals Research', **1**, Ed R F Bunsah, J Wiley, (New York) 1967.
6. J Pousey, A Royez and J P Georges, Rev Met 1959, **56**, 215.
7. E C Perryman, Polarized Light in Metallography, Ed G K Cann and R J Bradshaw, Academic Press (New York) 1952,
8. D McLean, J Inst Metals 1952, **81**, 121.
9. G Nomarski and A R Weill, Rev Met 1955, **52**, 121.
10. S Tolansky, Surface Microtopography, Interscience (New York) 1962.
11. R E Smallman and K H G Ashbee, Modern Metallography, Pergamon (London) 1966, p 19.
12. D Hull, PhD thesis, University of Wales, Cardiff, 1956.

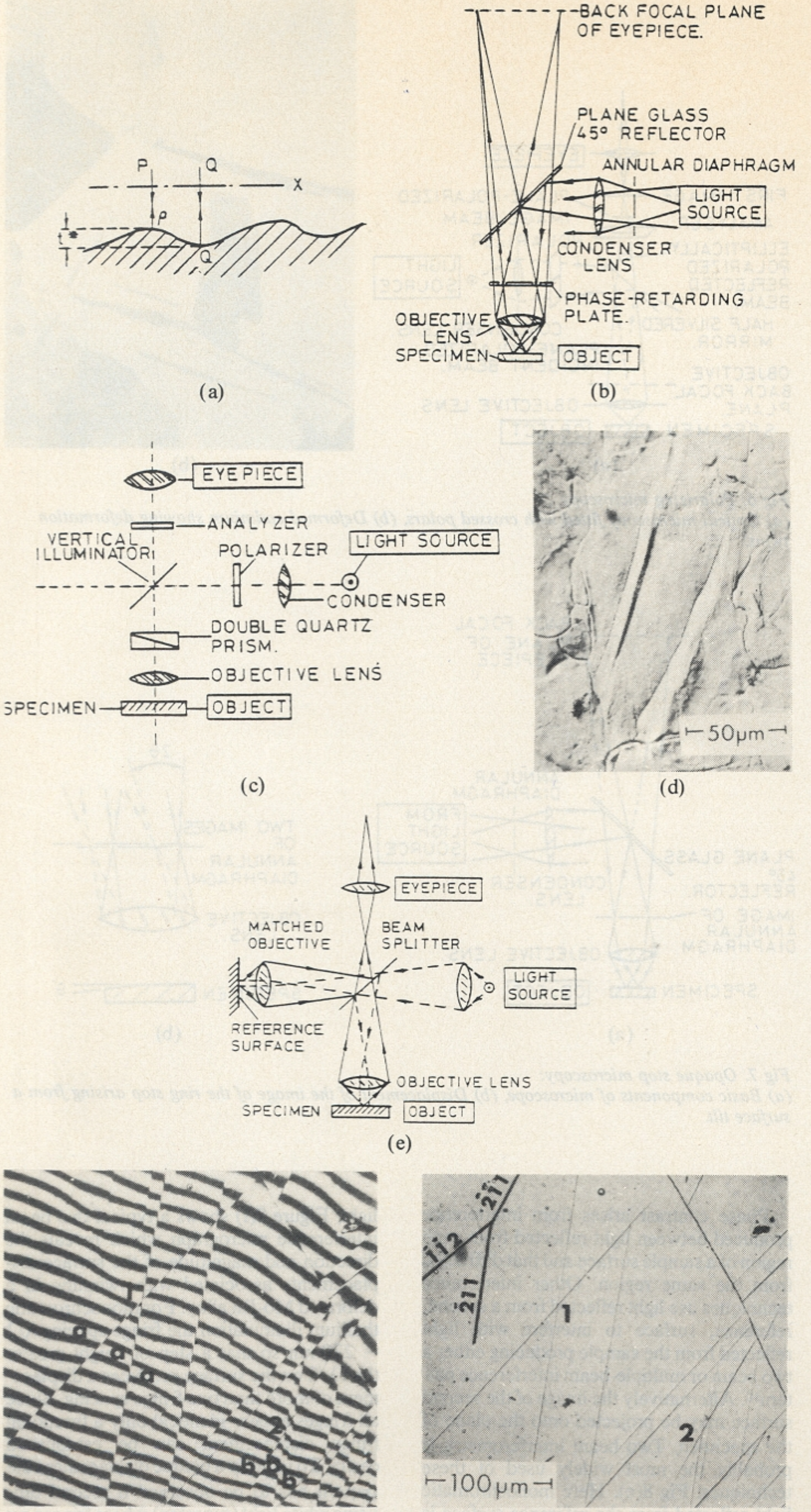

Fig 8. Phase and interference microscopy:
(a) Phase contrast due to reflecting dephasing object, (b) Phase contrast microscope, (c) The Nomarski reflection interference microscope; principle feature beam splitting prism, (d) Surface relief due to ferrite precipitation in a low alloy steel (Nomarski) (Courtesy D Swindon).
Two beam interference microscopy:
(e) Optical system, (f) {112} twins formed in deformed α phase Mo-10% Ru alloy, the interferogram is taken using monochromatic thallium illumination and shows the simple tilt displacement (a,a,a) of the surface associated with the twinning shear.

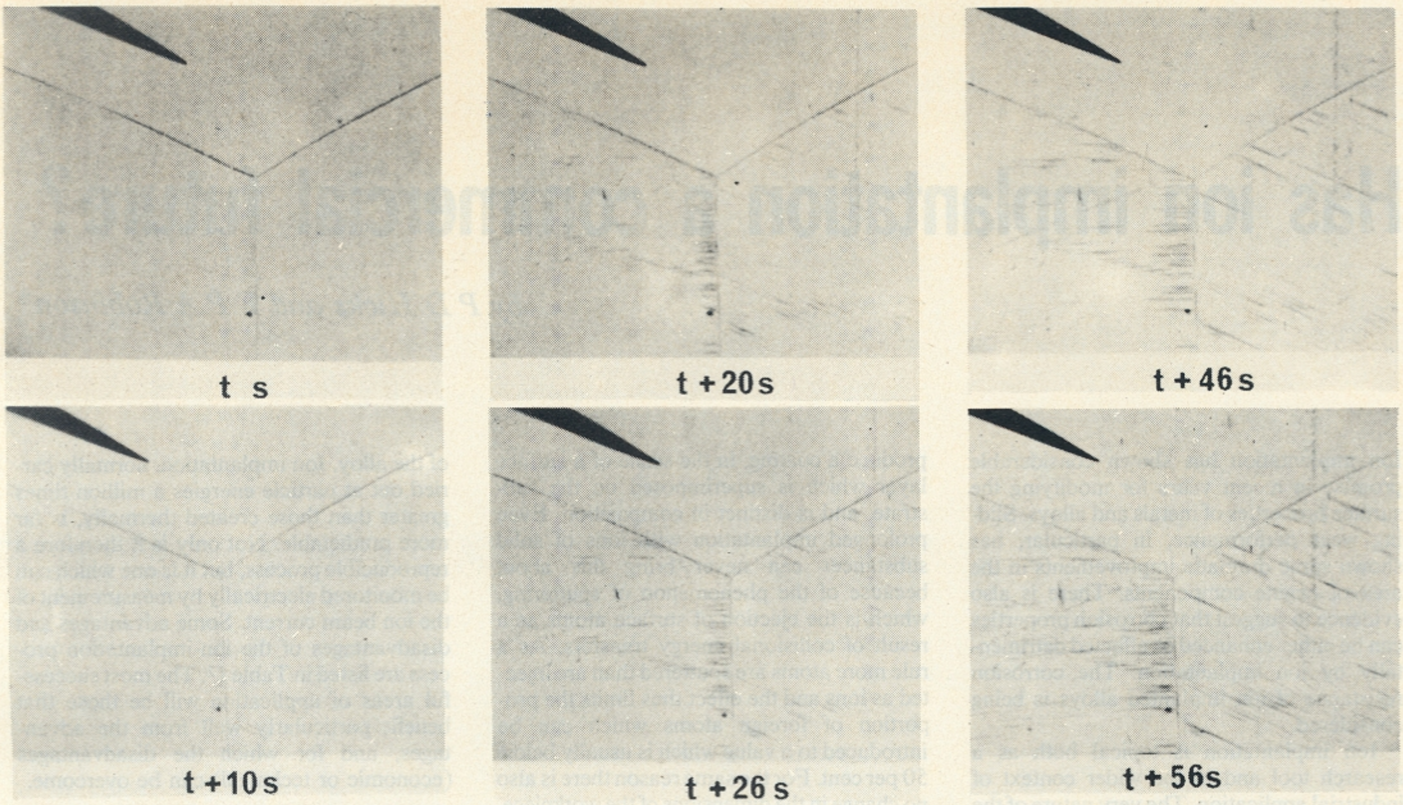

Fig 9. Hot stage microscopy: Growth of Widmanstätten α rods during isothermal ageing the metastable B' phase in a Cu-40% Zn alloy using a hot stage microscope (Ciné film sequence).